

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371Attorney's Docket Number 05725 0852  
Customer No.: 22,852

U.S. APPLICATION NO.

RECEIVED  
097763085

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|---|---|--|
| INTERNATIONAL APPLICATION NO.<br>PCT/FR00/01576   | INTERNATIONAL FILING DATE<br>June 8, 2000<br><i>10 MAY 2001</i><br><i>Legal Staff</i><br><i>International</i> | PRIORITY DATE CLAIMED<br>June 18, 1999 |
| TITLE OF INVENTION<br>AQUEOUS SOLID GEL COMPOSITIONS COMPRISING A HYDROPHILIC GELLING AGENT, A CELLULOSE DERIVATIVE AND PIGMENTS AND/OR MOTHER-OF PEARLS AND USES THEREOF |   |  |
| APPLICANT(S) FOR DO/EO/US<br>Isabelle BARA  |   |  |

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information

1.  This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2.  This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3.  This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4.  The US has been elected by the expiration of 19 months from the priority date (Article 31).
5.  A copy of the International Application as filed (35 U.S.C. 371 (c)(2)).
  - a.  is attached hereto (required only if not communicated by the International Bureau).
  - b.  has been communicated by the International Bureau.
  - c.  is not required, as the application was filed with the United States Receiving Office (RO/US).
6.  An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)).
  - a.  is attached hereto.
  - b.  has been previously submitted under 35 U.S.C. 154 (d)(4).
7.  Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3)).
  - a.  are attached hereto (required only if not communicated by the International Bureau).
  - b.  have been communicated by the International Bureau.
  - c.  have not been made; however, the time limit for making such amendments has NOT expired.
  - d.  have not been made and will not be made.
8.  An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9.  An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10.  An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

## Items 11 to 20 below concern document(s) or information included:

11.  Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12.  An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13.  A FIRST preliminary amendment.
14.  A SECOND or SUBSEQUENT preliminary amendment.
15.  A Substitute specification.
16.  A change of power of attorney and/or address letter.
17.  A computer-readable form of the sequence listing in accordance with PCT Rule 13ter 2 and 35 U.S.C. 1821-1825.
18.  A second copy of the published international application under 35 U.S.C. 154 (d)(4).
19.  A second copy of the English language translation of the international application 35 U.S.C. 154 (d)(4)
20.  Other items or information:
  - a.  Copy of cover page of International Publication No. WO 00/78856
  - b.  Copy of Notification of Missing Requirements.

|   |              |  |   |
|---|--------------|--|---|
| U.S. APPLICATION NO. 09/1763085 (5)   |              | INTERNATIONAL APPLICATION NO. PCT/FR00/01576 | ATTORNEY'S DOCKET NUMBER<br>05725 0852                        |
| 21. <input checked="" type="checkbox"/> The following fees are submitted:   |              |  | CALCULATIONS PTO USE ONLY                                     |
| <b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</b>   |              |  |   |
| Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... |              |  | \$1000.00   |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO .....   |              |  | \$860.00  |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search fee (37 CFR 1.445(a)(2)) paid to USPTO .....  |              |  | \$710.00  |
| International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) .....   |              |  | \$690.00  |
| International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33 (1)-(4) .....  |              |  | \$100.00  |
| <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>   |              |  | \$860.00  |
| Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e))  |              |  | <input type="checkbox"/> 20 <input type="checkbox"/> 30    \$ |
| CLAIMS  | NUMBER FILED | NUMBER EXTRA                                 | RATE  |
| Total Claims  | 29           | -20 = 9                                      | x \$18.00 \$162.00  |
| Independent Claims  | 1            | -3 = 0                                       | x \$80.00 \$  |
| MULTIPLE DEPENDENT CLAIM(S) (if applicable)   |              |  | +\$270.00 \$270.00  |
| <b>TOTAL OF THE ABOVE CALCULATIONS =</b>  |              |  | \$1292.00   |
| <input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by ½   |              |  | \$  |
| <b>SUBTOTAL =</b>   |              |  | \$1292.00   |
| Processing fee of <b>\$130.00</b> for furnishing the English translation later than months from the earliest priority date (37 CFR 1.492(f)).   |              |  | <input type="checkbox"/> 20 <input type="checkbox"/> 30    \$ |
| <b>TOTAL NATIONAL FEE =</b>   |              |  | \$1292.00   |
| Fee for recording the enclosed assignment (37 CFR 1.21 (h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property                        |              |  | +\$   |
| <b>TOTAL FEES ENCLOSED =</b>  |              |  | \$1292.00   |
|   |              |  | Amount to be refunded \$                                      |
|   |              |  | charged \$  |

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- a.  A check in the amount of \$ 1292.00 to cover the above fees is enclosed.
- b.  Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_ to cover the above fees.
- c.  A duplicate copy of this sheet is enclosed.
- d.  The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 06-0916. A duplicate copy of this sheet is enclosed.
- d.  Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status

**SEND ALL CORRESPONDENCE TO:**

Finnegan, Henderson, Farabow, Garrett & Dunner, L.L.P.  
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EFC/FPD/dvz  
DATED: February 16, 2001

  
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Ernest F. Chapman/25,961  
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PATENT

Attorney Docket No. 5725.0852

Customer No. 22,852

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re National Stage of International )  
Application No.: PCT/FR00/01576 of: )  
Isabelle BARA et al. )  
Application No.: Unassigned ) Group Art Unit: Unassigned  
PCT Filed: June 8, 2000 ) Examiner: Unassigned  
National Stage Entry: February 16, 2001 )  
)

For: SOLID AQUEOUS GEL COMPRISING A HYDROPHILIC GELLING  
AGENT, A CELLULOSE DERIVATIVE AND AT LEAST ONE PIGMENT  
AND/OR PEARLESCENT AGENT, AND METHODS FOR ITS USE (As  
Amended)

**BOX PCT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

**PRELIMINARY AMENDMENT**

Prior to examination of the above-identified application, please amend this  
application as follows:

**IN THE TITLE**

Please cancel the existing title and insert therefor --SOLID AQUEOUS  
GEL COMPRISING A HYDROPHILIC GELLING AGENT, A CELLULOSE

DERIVATIVE AND AT LEAST ONE PIGMENT AND/OR PEARLESCENT  
AGENT, AND METHODS FOR ITS USE --.

**IN THE CLAIMS:**

Please cancel claims 1-26 without prejudice or disclaimer and replace them with new claims 27-83 as follows:

-- 27. A solid aqueous gel comprising: i) at least one hydrophilic gelling agent, ii) at least one cellulose derivative, and iii) a pulverulent phase comprising at least one component chosen from pigments and pearlescent agents, wherein the combination of the hydrophilic gelling agent and the cellulose derivative is present in the gel in an amount ranging up to 20% by weight, relative to the total weight of the gel.

28. A gel according to claim 27, wherein the at least one hydrophilic gelling agent is chosen from polysaccharides, protein derivatives, synthetic and semisynthetic gels of the polyesters, polyacrylates, polymethacrylates, and derivatives thereof.

29. A gel according to claim 28, wherein the synthetic and semisynthetic gels of the polyesters are sulfonic synthetic and semisynthetic gels of the polyester.

30. A gel according to claim 28, wherein the at least one hydrophilic gelling agent is a polysaccharide chosen from:

- extracts of algae,
  - exudates of microorganisms,
  - fruit extracts,
  - gelling agents of animal origin,
  - polysaccharides possessing a side chain and 6 neutral sugars, and
  - mixtures thereof.
31. A gel according to claim 30, wherein the extracts of algae are chosen from agar, carrageenans, and alginates.
32. A gel according to claim 31, wherein the alginates are chosen from alginates of sodium and calcium.
33. A gel according to claim 30, wherein the exudates of microorganisms are chosen from xanthan gum, derivatives of xanthan gum, and gellan gum.
34. A gel according to claim 30, wherein the fruit extracts are chosen from pectins.
35. A gel according to claim 30, wherein the gelling agents of animal origin are chosen from derivatives of bovine and fish protein.
36. A gel according to claim 35, wherein the gelling agents of animal origin are chosen from caseinates and gelatin of bovine and fish origin.
37. A gel according to claim 28, wherein the at least one hydrophilic gelling agent is chosen from gellan and carrageenans.

38. A gel according to claim 27, wherein the at least one hydrophilic gelling agent is present in an amount ranging from 0.1% to 19.9% by weight, relative to the total weight of the gel.

39. A gel according to claim 38, wherein the at least one hydrophilic gelling agent is present in an amount ranging from 0.2% to 10% by weight, relative to the total weight of the gel.

40. A gel according to claim 27, wherein the at least one cellulose derivative is chosen from cellulose, carboxymethyl cellulose, hydroxypropyl cellulose, methyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose, and celluloses which are modified by grafting an alkyl group.

41. A gel according to claim 40, wherein the at least one cellulose derivative is carboxymethyl cellulose.

42. A gel according to claim 27, wherein the at least one cellulose derivative is present in an amount ranging from 0.1% to 10%, by weight relative to the total weight of the gel.

43. A gel according to claim 27, wherein the pigments are chosen from titanium; zirconium and cerium dioxides; zinc, iron and chromium oxides; nanotitaniums; ferric blue; carbon black; calcium, barium, aluminium and zirconium salts; acid dyes; azo dyes; anthraquinone dyes; pigments coated with silicone compounds; pigments coated with polymers; pigments coated with fluorinated compounds; and mixtures thereof.

44. A gel according to claim 43, wherein the acid dyes are chosen from halo-acid dyes.
45. A gel according to claim 43, wherein the pigments coated with silicone compounds are chosen from polydimethylsiloxanes.
46. A gel according to claim 43, wherein the pigments coated with polymers are chosen from polyethylenes.
47. A gel according to claim 27, wherein at least one pigment is present in an amount ranging up to 40% by weight, relative to the total weight of the gel.
48. A gel according to claim 47, wherein at least one pigment is present in an amount ranging from 0.1% to 30% by weight, relative to the total weight of the gel.
49. A gel according to claim 48, wherein at least one pigment is present in an amount ranging from 1% to 20% by weight, relative to the total weight of the gel.
50. A gel according to claim 27, wherein the pearlescent agents are chosen from natural pearl, mica coated with titanium oxide, mica coated with iron oxide, natural pigment, bismuth oxychloride, and colored mica-titanium.
51. A gel according to claim 27, wherein at least one pearlescent agent is present in an amount ranging up to 40% by weight, relative to the total weight of the gel.
52. A gel according to claim 51, wherein at least one

pearlescent agent is present in an amount ranging from 0.1% to 30% by weight, relative to the total weight of the gel.

53. A gel according to claim 52, wherein at least one pearlescent agent is present in an amount ranging from 1% to 20% by weight, relative to the total weight of the gel.

54. A gel according to claim 27, wherein the pulverulent phase is present in an amount ranging from 0.1% to 40%, by weight relative to the total weight of the gel.

55. A gel according to claim 54, wherein the pulverulent phase is present in an amount ranging from 0.1% to 20% by weight, relative to the total weight of the gel.

56. A gel according to claim 27, further comprising at least one filler.

57. A gel according to claim 56, wherein the at least one filler is chosen from talc, mica, silica, kaolin, Nylon powder, poly- $\beta$ -alanine powder, polyethylene powder, Teflon, lauroyllysine, starch, boron nitride, bismuth oxychloride, tetrafluoroethylene polymer powders, polymethyl methacrylate powders, polyurethane powders, polystyrene powders, polyester powders, synthetic hollow microspheres, microsponges, microbeads of silicone resin, zinc and titanium oxides, zirconium and cerium oxides, precipitated calcium carbonate, magnesium carbonate and hydrocarbonate, hydroxyapatite, hollow silica microspheres, glass and ceramic microcapsules, metallic soaps derived

from organic carboxylic acids comprising from 8 to 22 carbon atoms, the compounds  $\text{SiO}_2/\text{TiO}_2/\text{SiO}_2$ ,  $\text{TiO}_2/\text{CeO}_2/\text{SiO}_2$  and  $\text{TiO}_2/\text{ZnO/talc}$ , and polymers of polyethylene terephthalate/ polymethacrylate in the form of flakes.

58. A gel according to claim 57, wherein the metallic soaps derived from organic carboxylic acids comprise from 12 to 18 carbon atoms.

59. A gel according to claim 57, wherein the metallic soaps derived from organic carboxylic acids are chosen from zinc, magnesium and lithium stearate, zinc laurate, and magnesium myristate.

60. A gel according to claim 56, wherein the at least one filler is present in an amount ranging up to 60% by weight, relative to the total weight of the gel.

61. A gel according to claim 60, wherein the at least one filler is present in an amount ranging from 0.1% to 40% by weight, relative to the total weight of the gel.

62. A gel according to claim 61, wherein the at least one filler is present in an amount ranging from 1% to 20% by weight, relative to the total weight of the gel.

63. A gel according to claim 27, further comprising at least one salt.

64. A gel according to claim 63, wherein the at least one salt is chosen from calcium; magnesium and strontium nitrate; calcium and magnesium borate; calcium, sodium, magnesium, strontium, neodymium and manganese

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chloride; magnesium and calcium sulphate; and calcium and magnesium acetate.

65. A gel according to claim 64, wherein the at least one salt is magnesium chloride.

66. A gel according to claim 63, wherein the at least one salt is present in an amount ranging from 0.01% to 2% by weight, relative to the total weight of the gel.

67. A gel according to claim 66, wherein the at least one salt is present in an amount ranging from 0.1% to 1% by weight, relative to the total weight of the gel.

68. A gel according to claim 27, further comprising a cosmetically or physiologically acceptable medium.

69. A gel according to claim 27, further comprising a water chosen from floral water, mineral water, and thermal water.

70. A gel according to claim 69, wherein the floral water is cornflower water.

71. A gel according to claim 69, wherein the water is present in an amount ranging up to 99.8% by weight, relative to the total weight of the gel.

72. A gel according to claim 71, wherein the water is present in an amount ranging from 20% to 99% by weight, relative to the total weight of the gel.

73. A gel according to claim 27, further comprising at least one

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water-soluble colorant.

74. A gel according to claim 73, wherein the at least one water-soluble colorant is chosen from the disodium salt of ponceau, the disodium salt of alizarin green, quinoline yellow, the trisodium salt of amaranth, the disodium salt of tartrazine, the monosodium salt of rhodamine, the disodium salt of fuchsin, and xanthophyll.

75. A gel according to claim 27, further comprising at least one solvent chosen from ethanol, isopropanol, propylene glycol, butylene glycol, dipropylene glycol, diethylene glycol, and glycol ethers.

76. A gel according to claim 75, wherein the glycol ethers are chosen from (C<sub>1</sub>-C<sub>4</sub>) alkyl ethers of mono-, di-, and tripropylene glycol and from mono-, di-, and triethylene glycol.

77. A gel according to claim 27, further comprising at least one compound chosen from antioxidants, preservatives, hydrophilic cosmetic and pharmaceutical active agents, moisturizers, vitamins, self-tanning compounds, sunscreens, and perfumes.

78. A gel according to claim 27, wherein said gel is free of liquid fatty substances.

79. A gel according to claim 27, wherein said gel is free of a fatty phase.

80. A make-up product for the skin or the keratinous fibres comprising a solid aqueous gel comprising: i) at least one hydrophilic gelling

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agent, ii) at least one cellulose derivative, and iii) a pulverulent phase comprising at least one component chosen from pigments and pearlescent agents, wherein the combination of the hydrophilic gelling agent and the cellulose derivative is present in the gel in an amount ranging up to 20% by weight, relative to the total weight of the gel.

81. A make-up product for the body, a foundation, an eyeshadow, a blusher, a concealer, a lipstick, a pencil for the contour of the lips, a mascara, a pencil for the contour of the eyes, a dyeing or make-up stick for locks of hair comprising a solid aqueous gel comprising: i) at least one hydrophilic gelling agent, ii) at least one cellulose derivative, and iii) a pulverulent phase comprising at least one component chosen from pigments and pearlescent agents, wherein the combination of the hydrophilic gelling agent and the cellulose derivative is present in the gel in an amount ranging up to 20% by weight, relative to the total weight of the gel.

82. A method for applying make-up to the skin and/or the keratinous fibres, comprising applying to the skin and/or the keratinous fibres, a solid aqueous gel comprising: i) at least one hydrophilic gelling agent, ii) at least one cellulose derivative, and iii) a pulverulent phase comprising at least one component chosen from pigments and pearlescent agents, wherein the combination of the hydrophilic gelling agent and the cellulose derivative is present in the gel in an amount ranging up to 20% by weight, relative to the total weight of the gel.

83. A method for applying make-up to the skin and/or the keratinous fibers, comprising applying to the skin and/or the keratinous fibres, a make-up product for the skin or the keratinous fibres comprising a solid aqueous gel comprising: i) at least one hydrophilic gelling agent, ii) at least one cellulose derivative, and iii) a pulverulent phase comprising at least one component chosen from pigments and pearlescent agents, wherein the combination of the hydrophilic gelling agent and the cellulose derivative is present in the gel in an amount ranging up to 20% by weight, relative to the total weight of the gel.

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Remarks

Claims 1-26 have been cancelled without prejudice or disclaimer and replaced with new claims 27-83. New claims 27-83 have been added to more particularly point out and distinctly claim that which Applicants consider to be their invention. New claims 27-83 are not intended to be, nor believed to be, any more narrow than original claims 1-26. Support for each of the new claims be found throughout the specification and claims of the original application as filed. Accordingly, no new matter has been added.

If the Examiner believes a telephone conference would be helpful in advancing the prosecution of this application, the Examiner is respectfully urged to contact Applicants' undersigned representative at (202) 408-4193.

Please grant any extensions of time required to enter this Preliminary  
Amendment and charge any additional required fees to our Deposit Account  
06-0916.

Respectfully submitted,

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By: Michele C. Bosch  
Michele C. Bosch  
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SOLID AQUEOUS GEL COMPRISING A HYDROPHILIC GELLING AGENT, A CELLULOSE DERIVATIVE AND PIGMENTS AND/OR PEARLESCENT AGENTS AND ITS USES

5 The present invention relates to a solid aqueous gel, as well as its use in the cosmetics field, in particular as a make-up and/or health care product for the skin and/or the mucous membranes and/or the keratinous fibres.

Products which exist in solid form are known in the cosmetics industry. As products of this type, there may be mentioned, for example, in the make-up field, eyeshadow, foundation or lip sticks; in the field of skin or lip care, pencils for repairing the lips, depigmenting, make-up-removing or moisturizing sticks; in the field of hygiene, deodorant sticks, foam sticks or cakes for shaving or for washing the skin.

It is indeed particularly advantageous to have products in the form of sticks since such products are very convenient to use, they are easy to carry and there is no risk of the product running.

Moreover, make-up products are fairly generally formulated on the basis, on the one hand, of a fatty phase because of considerations of comfort and 25 smoothness and, on the other hand, of a pulverulent phase which provides the desired colour. This pulverulent phase may comprise pigments and/or fillers

and/or pearlescent agents. The fatty phase generally comprises waxes and/or oils and/or pasty compounds.

However, formulated sticks based on waxes have certain disadvantages: they have a greasy

- 5 character which is not appreciated by users and they lack freshness on application. In addition, it is difficult to introduce hydrophilic active agents therein.

Efforts are therefore being increasingly made

- 10 to produce make-up sticks comprising an aqueous phase in the highest concentration possible. It is of course known to produce nongreasy sticks such as deodorant sticks, but the latter are often formulated with soaps such as stearates of sodium hydroxide or of  
15 triethanolamine as gelling agent and they thus have a basic pH which is aggressive for the skin and is poorly compatible with the make-up pigments.

- Moreover, the presence of pigments or of  
pearlescent agents in aqueous sticks causes problems of  
20 disintegration of the product, that is to say of collecting of the product: indeed, these pigments and/or these pearlescent agents, which are essential for make-up products to provide colour, are solid particles which are not solubilized in the final  
25 composition. Because of this, they may interfere with the stability of the latter.

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However, it is essential, for a make-up product in particular, that the product is collected in an ideal manner, that is to say allows, in a simple manner, by means of the finger or a sponge or even 5 directly on the skin of the body for example, not only the appropriate quantity of product (not too much so as not to unnecessarily waste the product but enough to allow a make-up effect) to be collected but also the integrity of the product to be preserved at the time it 10 is collected: the product should not be broken through a phenomenon of shearing but the whole product should be collected with the pigments and/or the pearlescent agents, and/or optionally the fillers, which provide the make-up function. It is only on this condition that 15 the application of the product can be made homogeneously and the make-up obtained will be uniform.

Stable solid aqueous gels comprising pigments are known; but these products are generally impossible to disintegrate; they are often brittle; it is 20 therefore impossible to collect the product on the finger or on a sponge. Such products do not allow a homogeneous film to be deposited on the skin.

Products which can be disintegrated also exist but they are, in this case, too soft and sheared 25 during repeated applications or exhibit phenomena of syneresis over time, that is to say that the liquid portion exuded and the product has two phases: a solid

phase which cannot disintegrate and a liquid phase. The product can no longer perform its function, namely acting as make-up, since it is impossible to collect the pigments on the finger or on a sponge.

5       Aqueous rigid gels are described in the documents WO-A-97/17055 and WO-A-97/17053. However, these gels require the use of a fairly high concentration of gelling agent and involve a special preparation technique, extrusion. In addition, the  
10 sticks described in the document WO-A-97/17055, because of the high concentration of gelling agent, lack freshness and smoothness during application to the skin, and those described in the document WO-A-97/17053 have to be hydrated at the time of use. Furthermore,  
15 these gels often become brittle over time and no longer exhibit a good disintegration level.

Moreover, the document EP-A-803245 describes aqueous solid compositions containing thermoreversible polysaccharides, a humectant and a powdery phase which  
20 may comprise pigments. However, such gels do not have a satisfactory disintegration level.

Accordingly, the need remains for a solid aqueous gel which does not have the disadvantages of the prior art.

25       The Applicant has discovered, unexpectedly, that by combining a particular hydrophilic thickener, namely a cellulose derivative, with a hydrophilic

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gelling agent, and doing so in defined proportions, it is possible to produce solid aqueous gels comprising pigments and/or pearlescent agents, these gels being moreover homogeneous and stable, and being capable of 5 disintegrating easily on the finger or on a sponge or directly on the skin of the face or of the body.

This result is surprising; indeed, the combination of a thickener with a gelling agent usually contributes towards increasing the hardness of a gel 10 which indeed becomes solid, but also crumbly and brittle and often too hard and does not allow good application and low disintegration. The combination claimed below, with a particular hydrophilic thickener such as a cellulose derivative, makes it possible to 15 obtain a solid composition having ideal properties both in terms of stability and disintegration.

The present invention therefore relates to a solid aqueous gel, characterized in that it comprises i) at least one hydrophilic gelling agent, ii) at least 20 one cellulose derivative and iii) a pulverulent phase comprising at least one pigment and/or one pearlescent agent, the combination of the hydrophilic gelling agent and of the cellulose derivative being present in the gel in an amount of less than or equal to 20% by weight 25 relative to the total weight of the gel.

The gels of the invention exhibit excellent application and disintegration qualities. In

particular, by virtue of the combination according to the invention, a disintegration level is obtained, for an equivalent hardness, which is greater than that of known sticks. The product is easy to collect, this can  
5 be achieved directly on the body or with the finger or with a sponge, by removing a sufficient quantity of product, which is then easy to apply to the skin in a homogeneous manner without requiring preliminary wetting. The make-up obtained is uniform and  
10 homogeneous.

These gels are stable over time and with regard to temperature. Thus, after having been stored for two months at room temperature or at 45°C, they exhibit no phenomenon of syneresis (exudation) or phase separation; their appearance and their hardness did not vary.

The gels according to the invention do not become exuded, even for low levels of gelling agent, and they do not necessarily require the use of a special preparation technique. They offer upon application a feeling of great freshness while preserving good cosmetic properties, in particular properties of smoothness.

The subject of the present invention is also  
25 a make-up product for the skin or the keratinous fibres  
comprising a gel as defined above.

The subject of the present invention is also a method for applying make-up to the skin and/or the keratinous fibres, consisting in applying to the skin and/or the keratinous fibres a make-up gel and/or  
5 product as defined above.

For the purposes of the present invention, the expression solid gel is understood to mean a gel having a hardness which is defined by a maximum load before breaking ranging from 5 to 50 grams, at room  
10 temperature (20-25°C), after penetration by a stainless-steel rotor 2 mm in diameter in the matrix of the composition at a thickness of 1 mm at a speed of 1 mm/s and withdrawal of the said rotor from the matrix of the composition at a speed of 2 mm/s, the maximum  
15 load before breaking being measured with a texture analyser of the "TAXT2" type marketed by the company RHEO.

Preferably still, the maximum load before breaking ranges from 7 to 40 g.

20 The gel according to the invention comprises a hydrophilic gelling agent. The expression gelling agent is understood to mean a compound which, in the presence of a solvent, creates intermacromolecular bonds which are strong to a greater or lesser degree,  
25 thus inducing a three-dimensional network which immobilizes the said solvent.

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This hydrophilic gelling agent may be chosen from polysaccharides, protein derivatives, synthetic or semisynthetic gels of the polyester, in particular sulphonic, type, polyacrylates or polymethacrylates and 5 derivatives thereof.

Among the polysaccharides, there may be mentioned:

- extracts of algae such as agar, carrageenans (iota, kappa, lambda), alginates, in 10 particular of Na or Ca;
- exudates of microorganisms such as xanthan gum and its derivatives such as the product sold under the trade name "Rhéosan" by the company Rhodia Chimie, gellan;
- 15 - fruit extracts such as pectins;
- gelling agents of animal origin such as bovine or fish protein, in particular gelatin, derivatives, caseinates;
- polysaccharides possessing a side chain 20 and 6 neutral sugars as described in the document FR-A-2759377;
- and mixtures thereof.

Among the synthetic or semisynthetic gels, there may be mentioned the copolymers described in 25 application FR-A-2 760 643.

Preferably, the hydrophilic gelling agent is chosen from polysaccharides, preferably still from gellan, carrageenans and mixtures thereof.

As products which are particularly suitable 5 for the invention, there may be mentioned the gellan gum sold under the trade name "Kelcogel F" by the company NUTRASWEET-KELCO or alternatively the iodocarrageenan sold under the trade names "Seaspen PF 357" or "Viscarin SD 389" by the company FMC.

10 The hydrophilic gelling agent is present in the gel according to the invention at a concentration which makes it possible to obtain, in combination with the cellulose derivative, the hardness and the consistency which are suitable for an ideal 15 disintegration. The hydrophilic gelling agent is preferably present in the gel according to the invention at a concentration which may range from 0.1 to 19.9%, preferably still from 0.2 to 10%, by weight relative to the total weight of the gel.

20 The gel according to the invention also comprises a cellulose derivative. This derivative may for example be chosen from:

- cellulose, carboxymethyl cellulose,  
hydroxypropyl cellulose, methyl cellulose,  
25 hydroxypropyl methyl cellulose or hydroxyethyl cellulose as well as celluloses which are modified in particular by grafting an alkyl group.

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Preferably the cellulose derivative is carboxymethyl cellulose.

As a cellulose derivative which is particularly suitable for the invention, there may be  
5 mentioned:

- sodium carboxymethyl cellulose sold under the trade name "Blanose 7 LF" by the company Aqualon,
- hydroxyethyl cellulose sold under the trade name "Natrosol 250 HHR" by the company Aqualon,
- 10 - hydroxypropyl cellulose sold under the trade name "KLUCEL H" by the company Aqualon,
- cetyl hydroxyethyl cellulose sold under the trade name "Polysurf 67" by the company AQUALON.

The cellulose derivative is present in the  
15 gel according to the invention at a concentration which makes it possible to obtain, in combination with the hydrophilic gelling agent, the hardness and the consistency which are appropriate for an ideal disintegration. Preferably, the cellulose derivative is  
20 present in the gel according to the invention at a concentration which may range from 0.1 to 10% relative to the total weight of the gel.

The combination of the hydrophilic gelling agent and of the cellulose derivative is present in the  
25 gel according to the invention at an overall level which makes it possible to obtain the hardness and the consistency which are appropriate for an ideal

disintegration, namely a hardness, measured as described above, defined by a load ranging from 5 to 50 g and preferably from 7 to 40 g. Thus, this overall level ranges up to 20% by weight relative to the total 5 weight of the gel.

For example, for a gellan level of 0.5% by weight relative to the total weight of the gel, a carboxymethyl cellulose level of between 0.5 and 1.8%, and even better between 0.5 and 1.3% by weight relative 10 to the total weight of the gel makes it possible to obtain a gel having the hardness and the consistency which are ideal for good disintegration.

The gel according to the invention also comprises a pulverulent phase which comprises at least 15 one pigment and/or one pearlescent agent.

The term pigments should be understood to mean white or coloured, inorganic or organic particles which are insoluble in the medium and which are intended to colour and/or opacify the composition.

20 The pigments may be present in an amount of 0-40% by weight relative to the total weight of the gel, preferably in an amount of 0.1 to 30% and preferably still in an amount of 1-20%. They may be white or coloured, inorganic and/or organic, of the 25 usual or of a nanometric size. The expression nanometric size is understood to mean pigments whose mean particle size ranges from 5 to 100 nm.

There may be mentioned, among the inorganic pigments and nanopigments, titanium, zirconium or cerium dioxides, as well as zinc, iron or chromium oxides, nanotitaniums, ferric blue and/or mixtures

5 thereof. Among the organic pigments, there may be mentioned carbon black, and the lacquers commonly used to confer a make-up effect on the lips and on the skin, which are calcium, barium, aluminium or zirconium salts, acid dyes such as halo-acid, azo or

10 anthraquinone dyes and/or mixtures thereof.

The pigments may in particular be coated with silicone compounds such as PDMSs and/or with polymers, in particular polyethylenes, or alternatively with fluorinated compounds. There may thus be mentioned the  
15 SA pigments from Maprecos or the PI pigments from Myoshi.

The expression pearlescent agents should be understood to mean iridescent particles which reflect light.

20 The pearlescent agents may be present in the gel in an amount of 0-40% by weight, preferably in an amount of 0.1 to 30% and preferably still in an amount of 1-20% by weight.

Among the pearlescent agents which may be  
25 envisaged, there may be mentioned natural pearl, mica coated with titanium oxide, iron oxide, natural pigment

or bismuth oxychloride as well as coloured mica-titanium.

In general, the pulverulent phase comprises enough pigments and/or pearlescent agents to provide  
5 the desired make-up effect. Thus, preferably, the aqueous gel according to the invention is not transparent, that is to say that it is not possible to see the characters on a newspaper page through the gel. Preferably still it is not translucent, that is to say  
10 that it does not allow the passage of light.

The pulverulent phase preferably comprises from 0.1 to 40%, preferably still from 0.1 to 20% by weight, relative to the total weight of the gel, of pigments and/or pearlescent agents.

15 The pulverulent phase of the gels according to the invention may comprise, in addition, fillers.

The term fillers should be understood to mean colourless or white, inorganic or synthetic, lamellar or nonlamellar particles intended to give the  
20 composition body or stiffness, and/or the make-up smoothness, mattness and uniformity.

The fillers, which may be present in the gel in an amount of 0-60% by weight relative to the total weight of the gel, preferably in an amount of 0.1 to  
25 40%, preferably still 1-20%, may be inorganic or synthetic, lamellar or nonlamellar.

There may be mentioned talc, mica, silica, kaolin, Nylon powder, poly- $\beta$ -alanine powder and polyethylene powder, Teflon, lauroyllysine, starch, boron nitride, bismuth oxychloride, tetrafluoroethylene  
5 polymer powders, polymethyl methacrylate powders, polyurethane powders, polystyrene powders, polyester powders, synthetic hollow microspheres, microsponges, microbeads of silicone resin, zinc and titanium oxides, zirconium or cerium oxides, precipitated calcium  
10 carbonate, magnesium carbonate and hydrocarbonate, hydroxyapatite, hollow silica microspheres, glass or ceramic microcapsules, metallic soaps derived from organic carboxylic acids having from 8 to 22 carbon atoms, preferably from 12 to 18 carbon atoms, such as  
15 zinc, magnesium or lithium stearate, zinc laurate, magnesium myristate, the compounds  $\text{SiO}_2/\text{TiO}_2/\text{SiO}_2$ ,  $\text{TiO}_2/\text{CeO}_2/\text{SiO}_2$  or  $\text{TiO}_2/\text{ZnO}/\text{talc}$ , and polymers of polyethylene terephthalate/polymethacrylate in the form of flakes.

20 It is possible to modify the stiffness of the gels according to the invention by adding thereto one or more salts which will increase this stiffness. These salts may be chosen from mono-, di- or trivalent metal salts, and more particularly alkali and alkaline earth  
25 metal salts and in particular sodium, calcium or magnesium salts. The ions which constitute these salts may be chosen, for example, from carbonates,

bicarbonates, sulphates, glycerophosphates, borates, chlorides, nitrates, acetates, hydroxides, persulphates as well as the salts of  $\alpha$ -hydroxy acids (citrates, tartrates, lactates, malates) or of fruit acids, or  
5 alternatively the salts of amino acids (aspartate, arginate, glycocholate, fumarate). The quantity of salt may range from 0.01 to 2% and preferably from 0.1 to 1% of the total weight of the gel.

Preferably, the salt is chosen from calcium,  
10 magnesium or strontium nitrate, calcium or magnesium borate, calcium, sodium, magnesium, strontium, neodymium or manganese chloride, magnesium or calcium sulphate, calcium or magnesium acetate, and mixtures thereof. Preferably still, the salt is magnesium  
15 chloride.

The gels of the invention contain, in addition, a cosmetically or physiologically acceptable medium, that is to say a medium which is compatible with all keratinous materials such as the skin, nails,  
20 hair, eyelashes and eyebrows, mucous membranes and semimucous membranes, and any other skin area of the body and of the face.

The gels according to the invention may also comprise floral water such as cornflower water and/or  
25 mineral water such as VITTEL water, LUCAS water or LA ROCHE POSAY water and/or thermal water.

The gels according to the invention may also comprise water-soluble colorants chosen from the customary colorants in the field considered, such as the disodium salt of ponceau, the disodium salt of 5 alizarin green, quinoline yellow, the trisodium salt of amaranth, the disodium salt of tartrazine, the monosodium salt of rhodamine, the disodium salt of fuchsin, or xanthophyll.

Preferably, the gels according to the 10 invention comprise up to 99.8% by weight, preferably from 20 to 99% by weight, relative to the total weight of the gel, of water.

The gels according to the invention may also comprise solvents other than water, such as for example 15 primary alcohols such as ethanol and isopropanol, glycols such as propylene glycol, butylene glycol, dipropylene glycol, diethylene glycol, glycol ethers such as ( $C_1-C_4$ )alkyl ethers of mono-, di- or tripropylene glycol, mono-, di- or triethylene glycol, 20 and mixtures thereof.

The gels of the invention may comprise, in addition, any additional hydrophilic compound customarily used in the cosmetics field. These additional compounds may be chosen from antioxidants, 25 preservatives, hydrophilic cosmetic or pharmaceutical active agents, moisturizers, vitamins, self-tanning

compounds such as DHA, sunscreens, perfumes, and mixtures thereof.

These additional compounds may be present in the gel in an amount of 0-10% by weight.

5        Of course, persons skilled in the art will be careful to choose this or these possible additional compounds, and/or their quantity, in a manner such that the advantageous properties of the gel according to the invention are not, or not substantially, adversely  
10 affected by the envisaged addition.

Preferably, the gels according to the invention are free of liquid fatty substances such as oils. Preferably still, they are free of a fatty phase.

The gels according to the invention may be  
15 prepared according to conventional methods for preparing sticks, these methods being well known to persons skilled in the art.

The gels according to the invention may constitute make-up or health care products for the  
20 skin, in particular the body, the face and/or the scalp, or the keratinous fibres, in particular the hair, the nails, the eyelashes and/or the eyebrows, or the mucous membranes, in particular the lips. They can thus constitute make-up products for the body,  
25 foundations, eyeshadows, blushers, concealers, lipsticks, pencils for the contour of the lips,

mascaras, pencils for the contour of the eyes, dyeing or make-up sticks for locks of hair.

The invention is illustrated in greater detail in the following examples.

5 In the following examples, the quantities are given as a percentage by weight relative to the total weight of the composition.

EXAMPLE 1:

10 The Applicant produced the aqueous gel in the form of the following foundation stick:

|  |         |
|--|---------|
| - Gellan gum sold under the trade name "Kelcogel F" from NUTRASWEET-KELCO                        | 0.5%    |
| - Mg chloride  | 0.1%    |
| - Sodium carboxymethyl cellulose sold under the trade name "Blanose 7 LF" by the company Aqualon | 1.2%    |
| - Preservative   | qs      |
| - Pigments (iron oxides and titanium dioxide)  | 7%      |
| - Propylene glycol   | 7%      |
| - Water  | qs 100% |

This gel was prepared in the following manner: the water and the preservative were heated to 90°C, and then the gellan was incorporated with stirring. After waiting for 15 min, the carboxymethyl cellulose was incorporated with stirring, and then the pigmentary paste produced beforehand by mixing the pigments with propylene glycol.

The mixture was subsequently poured into a dish and then cooled. The whole is allowed to stand for 24 h at room temperature.

A stick is obtained which is very fresh upon 5 application, which can be used with a dry or wet sponge, which can be collected well and which can be easily and homogeneously applied to the skin.

The application of make-up to the skin with such a stick gives a natural and transparent result, 10 totally free of greasy effect.

The hardness of this gel, measured as described above, is 14 g.

**EXAMPLE 2:**

The Applicant produced the following aqueous 15 gel in the form of a stick for tattooing the body:

|  |         |
|--|---------|
| - Lambda-carrageenan sold under the trade name "Satia gum UTC 10" by FKW Biosystems              | 5%      |
| - Gellan gum sold under the trade name "Kelcogel F" from NUTRASWEET-KELCO                        | 0.5%    |
| - Sodium carboxymethyl cellulose sold under the trade name "Blanose 7 LF" by the company Aqualon | 1.2%    |
| - Preservative   | qs      |
| - Water  | qs 100% |
| - Pearlescent agents   | 5%      |

This stick was prepared in the same manner as in Example 1.

A stick is obtained which can be used directly on the skin, which disintegrates well so as to draw motifs or lines on the body.

The hardness of this stick, measured as 5 described above, is 5.6 g.

The Applicant also produced the gel of Example 2 without carboxymethyl cellulose: such a gel has a hardness of 7.4 g, but it disintegrates with difficulty.

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**CLAIMS**

1. Solid aqueous gel, characterized in that it comprises i) at least one hydrophilic gelling agent,  
5 ii) at least one cellulose derivative and iii) a pulverulent phase comprising at least one pigment and/or one pearlescent agent, the combination of the hydrophilic gelling agent and of the cellulose derivative being present in the gel in an amount of  
10 less than or equal to 20% by weight relative to the total weight of the gel.

2. Gel according to Claim 1, characterized in that the hydrophilic gelling agent is chosen from polysaccharides, protein derivatives, synthetic or  
15 semisynthetic gels of the polyester, in particular sulphonic, type, polyacrylates or polymethacrylates and derivatives thereof.

3. Gel according to Claim 2, characterized in that the hydrophilic gelling agent is a  
20 polysaccharide chosen from:

- extracts of algae such as agar, carrageenans, alginates, in particular alginates of Na or Ca;
- exudates of microorganisms such as  
25 xanthan gum and its derivatives or gellan gum;
- fruit extracts such as pectins;

- gelling agents of animal origin such as derivatives of bovine or fish protein, in particular of gelatin of bovine or fish origin or caseinates;

- polysaccharides possessing a side chain 5 and 6 neutral sugars;

- and mixtures thereof.

4. Gel according to Claim 3, characterized in that the hydrophilic gelling agent is chosen from gellan, carrageenans and mixtures thereof.

10 5. Gel according to any one of the preceding claims, characterized in that the hydrophilic gelling agent is present at a concentration ranging from 0.1 to 19.9%, preferably from 0.2 to 10%, by weight relative to the total weight of the gel.

15 6. Gel according to any one of the preceding claims, characterized in that the cellulose derivative is chosen from cellulose, carboxymethyl cellulose, hydroxypropyl cellulose, methyl cellulose, hydroxypropyl methyl cellulose or hydroxyethyl 20 cellulose as well as celluloses which are modified in particular by grafting an alkyl group.

7. Gel according to Claim 6, characterized in that the cellulose derivative is carboxymethyl cellulose.

25 8. Gel according to any one of the preceding claims, characterized in that the cellulose

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derivative is present at a concentration ranging from 0.1 to 10% relative to the total weight of the gel.

9. Gel according to any one of the preceding claims, characterized in that at least one 5 pigment is chosen from titanium, zirconium or cerium dioxides, zinc, iron or chromium oxides, nanotitaniums, ferric blue, carbon black, calcium, barium, aluminium or zirconium salts, acid dyes such as halo-acid, azo or anthraquinone dyes, pigments coated with silicone 10 compounds such as polydimethylsiloxanes and/or with polymers, in particular polyethylenes, or alternatively with fluorinated compounds, and/or mixtures thereof.

10. Gel according to any one of the preceding claims, characterized in that at least one 15 pigment is present in an amount ranging up to 40% by weight, preferably from 0.1 to 30% by weight, relative to the total weight of the gel.

11. Gel according to any one of the preceding claims, characterized in that at least one 20 pearlescent agent is chosen from natural pearl, mica coated with titanium oxide, iron oxide, natural pigment or bismuth oxychloride as well as coloured mica-titanium.

12. Gel according to any one of the 25 preceding claims, characterized in that at least one pearlescent agent is present in an amount ranging up to

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40% by weight, preferably from 0.1 to 30% by weight, relative to the total weight of the gel.

13. Gel according to any one of the preceding claims, characterized in that the pigment 5 and/or pearlescent agent is present in an amount ranging from 0.1 to 40%, preferably still from 0.1 to 20% by weight, relative to the total weight of the gel.

14. Gel according to any one of the preceding claims, characterized in that it comprises, 10 in addition, at least one filler.

15. Gel according to the preceding claim, characterized in that the filler is chosen from talc, mica, silica, kaolin, Nylon powder, poly- $\beta$ -alanine powder and polyethylene powder, Teflon, lauroyllysine, 15 starch, boron nitride, bismuth oxychloride, tetrafluoroethylene polymer powders, polymethyl methacrylate powders, polyurethane powders, polystyrene powders, polyester powders, synthetic hollow microspheres, microsponges, microbeads of silicone 20 resin, zinc and titanium oxides, zirconium or cerium oxides, precipitated calcium carbonate, magnesium carbonate and hydrocarbonate, hydroxyapatite, hollow silica microspheres, glass or ceramic microcapsules, metallic soaps derived from organic carboxylic acids 25 having from 8 to 22 carbon atoms, preferably from 12 to 18 carbon atoms, such as zinc, magnesium or lithium stearate, zinc laurate, magnesium myristate, the

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compounds  $\text{SiO}_2/\text{TiO}_2/\text{SiO}_2$ ,  $\text{TiO}_2/\text{CeO}_2/\text{SiO}_2$  or  $\text{TiO}_2/\text{ZnO/talc}$ , and polymers of polyethylene terephthalate/polymethacrylate in the form of flakes.

16. Gel according to Claim 14 or 15,  
5 characterized in that the fillers are present in an amount ranging up to 60% by weight, preferably from 0.1 to 40% by weight, relative to the total weight of the gel.

17. Gel according to any one of the  
10 preceding claims, characterized in that it comprises, in addition, a salt.

18. Gel according to the preceding claim, characterized in that the salt is chosen from calcium, magnesium or strontium nitrate, calcium or magnesium borate, calcium, sodium, magnesium, strontium, neodymium or manganese chloride, magnesium or calcium sulphate, calcium or magnesium acetate, and mixtures thereof.

19. Gel according to the preceding claim,  
20 characterized in that the salt is magnesium chloride.

20. Gel according to any one of the preceding claims, characterized in that it comprises, in addition, a cosmetically or physiologically acceptable medium.

- 25 21. Gel according to any one of the preceding claims, characterized in that it comprises, in addition, a water-soluble colorant.

22. Gel according to any one of the preceding claims, characterized in that it comprises, in addition, a solvent chosen from ethanol, isopropanol, propylene glycol, butylene glycol, 5 dipropylene glycol, diethylene glycol, glycol ethers, and mixtures thereof.

23. Gel according to any one of the preceding claims, characterized in that it comprises, in addition, an additional compound chosen from 10 antioxidants, preservatives, hydrophilic cosmetic or pharmaceutical active agents, moisturizers, vitamins, self-tanning compounds, sunscreens, perfumes, and mixtures thereof.

24. Make-up product for the skin or the 15 keratinous fibres, characterized in that it comprises a gel as defined in any one of Claims 1 to 23.

25. Product according to Claim 24, characterized in that it constitutes a make-up product for the body, a foundation, an eyeshadow, a blusher, a 20 concealer, a lipstick, a pencil for the contour of the lips, a mascara, a pencil for the contour of the eyes, a dyeing or make-up stick for locks of hair.

26. Method for applying make-up to the skin and/or the keratinous fibres, consisting in applying to 25 the skin and/or the keratinous fibres, a gel as defined in any one of Claims 1 to 23 and/or a product as defined in either of Claims 24 and 25.

#### DESCRIPTIVE ABSTRACT

SOLID AQUEOUS GEL COMPRISING A HYDROPHILIC GELLING  
AGENT, A CELLULOSE DERIVATIVE AND PIGMENTS AND/OR  
PEARLESCENT AGENTS AND ITS USES

The present invention relates to a solid aqueous gel, characterized in that it comprises i) at least one hydrophilic gelling agent, ii) at least one cellulose derivative and iii) a pulverulent phase comprising at least one pigment and/or one pearlescent agent, the combination of the hydrophilic gelling agent and of the cellulose derivative being present in the gel in an amount of less than or equal to 20% by weight relative to the total weight of the gel.

This gel may be used in stick or compact (waterpact) form and can constitute make-up products for the skin and/or the mucous membranes and/or the keratinous fibres. This gel has the advantage of being stable (absence of syneresis). It allows easy and homogeneous disintegration of the product and provides great freshness upon application.